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August 3, 2007

Mr. Michael Berkoff
Remedial Project Manager
United States Environmental Protection Agency
Region 5, Attn: SR-6J
77 W. Jackson Blvd.
Chicago, IL 60604-3590

Subject: Response to Comments on Quality Assurance Project Plan
12th Street Landfill, Kalamazoo River Superfund Site Operable Unit #04
Plainwell, Michigan

Dear Mr. Berkoff:

This letter is in response to the July 24, 2007, conditional approval letter from the United States Environmental Protection Agency (USEPA) to RMT, Inc. (RMT), which provided comments on the draft Multi-Area Quality Assurance Project Plan (QAPP) for the Emergency Response Activities at the Allied Paper, Inc./Portage Creek/Kalamazoo River Superfund Site. The draft Multi-Area QAPP was submitted by RMT to the USEPA on June 21, 2007, on behalf of Weyerhaeuser Company.

Each of the four USEPA comments is provided below in italics, along with responses and additional information, as applicable.

1. *QAPP Worksheet # 11: Second paragraph. Please provide a reference for the sampling and analytical protocol instead of "defined later in this document."*

   RMT will replace the current sentence, "Sampling and analytical protocols are defined later in this document," with, "Sampling protocols are detailed in the project sampling SOPs, which are listed in QAPP Worksheet #21. The project sampling SOPs are part of the Field Sampling Plan, which was submitted to the USEPA for this project. Analytical protocols are detailed in the analytical SOPs, which are listed in QAPP Worksheet #23 and attached to this Multi-Area QAPP. Information regarding analytical instrument calibration, maintenance, testing, and inspection are provided in QAPP Worksheets #24 and #25."

2. *QAPP Worksheet # 11: Last paragraph. Please put an estimate amount of samples which you are planning to collect and analyze to satisfy the project goal.*

   The number of samples that will be collected for this project is a function of the project duration. Since the project duration is dependent on a number of factors, including some factors that are outside of our control (*e.g.*, weather), the scope of the sampling program for this project has been defined by sampling frequency, rather than an estimate number of total samples. For example, samples of the effluent from the wastewater treatment system will be collected and analyzed for PCBs, total suspended solids, and total phosphorus twice per week for the duration of system operation.

   In response to this comment, RMT will insert the following sentences into QAPP Worksheet #11, "The sampling program is summarized in QAPP Worksheet #18 and in Table 3-1 of the Emergency Response Plan Design Report, which was submitted to the USEPA for this project. The total number of samples will depend on the project duration. Estimated durations for various phases of the project were provided in the

Project Schedule (Figure 7 of the Emergency Response Plan Design Report); however, the actual durations will vary based on field conditions."

3. *All Standard Operating Procedures (SOPs) from Weyerhaeuser Analysis and Testing (WATS) laboratory should be updated and resubmitted for review. Standard Operating Procedure (SOP) from this laboratory should be written in the requirement format according to EPA QA/G-6 document: www.epa.gov/quality/qs-docs/g6-final.pdf*

   Seven WATS SOPs were included in the draft Multi-Area QAPP. Two of these seven WATS SOPs were recently revised to reflect applicable comments provided by the USEPA on the draft QAPP that was submitted with the Plainwell Mill RI/FS Work Plan. These two revised SOPs are attached to this response letter (see Attachment 1). The USEPA has not provided specific comments on the other five SOPs; therefore, WATS' other SOPs have not been resubmitted.

   As directed in the above comment, the specified USEPA guidance document was reviewed to provide input to this response. This guidance document explicitly states that "SOPs should be organized to ensure ease and efficiency in use and to be specific to the organization which develops it. There is no one 'correct' format; and internal formatting will vary with each organization and with the type of SOP being written." (EPA QA/G-6, April 2007, pg 6). This introductory paragraph continues with suggestions that include formatting SOPs into logical steps and providing the amount of detail needed for the individual procedure. The final sentence in the introductory paragraph includes additional detail to assist users in organizing their SOPs.

   As suggested in the guidance, SOPs for WATS have been developed to meet the regulatory needs of their data users (often facilities) and still be consistent with other corporate initiatives like Total Quality Management and ISO certifications. Thus, the WATS SOPs meet both external and internal requirements. Multiple states, several USEPA regions, including Region 5, and independent ISO 14000 auditors have approved WATS' SOPs. They are also used throughout the organization and reflect company policies regarding commitment to quality and safety. Although the individual sections are somewhat different from those in the generalized guidance summary, the WATS SOPs provide essentially all of the pertinent information suggested in Section 4.1 of the guidance. The slight deviations from the guidance will be addressed, as described in right-most column of Table 1 (*i.e.*, Modifications Needed to Meet Objectives).

4. *QAPP Worksheets #34, 35, and 36. Data Verification/Validation. Please explain how the data review and verification will be organized in this project meet the Superfund requirements. A 100% laboratory data validation must be performed by an entity independent of the laboratory.*

   In accordance with Superfund requirements, validation of 100% of laboratory data will be performed by RMT, which is an entity independent of the laboratory. As described under "Documentation and Records" in QAPP Worksheet #14, WATS will prepare and provide full "Contract Laboratory Program (CLP)-like" data packages. These data packages will be provided to the RMT Data QA Manager, who will review and validate the data packages as described in Worksheets #34, #35, and #36 and in accordance with the USEPA Contract Laboratory National Functional Guidelines for Organic Data Review (October 1999).

If you have any questions regarding the information provided, please contact me at 262-879-1212.

Sincerely,

RMT, Inc.

James L. Hutchens
Senior Project Manager

cmk/attachments

cc: Jennifer Hale, Weyerhaeuser Company
Martin Lebo, Weyerhaeuser Company
Keith Krawczyk, MDEQ
Kathy Huibregtse, RMT, Inc.
Nate Weber, RMT, Inc.

Table 1
Comparison of EPA Guidelines and WATS SOP Format

EPA Guidelines General Elements for SOPs	Specific Objectives of Section	Applicable Weyerhaeuser Section	Modifications Needed to Meet Objectives
Title Page	Present identifying information and signature block	Title Block	No modifications to meet objective.
Table of Contents	Quick Reference especially if SOP is long	None	Each of Weyerhaeuser's analytical SOPs has 13 standard sections. Those sections will be identified on the cover sheet to Appendix 1-5.
Procedures	Present purpose, define unusual terms, denote sequential procedures, equipment needs, personnel qualifications and safety considerations to the level of detail appropriate to the procedure	For analytical SOPs, Sections 1 to 9 address procedures as listed below: 1. Scope 2. Summary of Procedure 3. Interferences 4. Estimate of Analytical time 5. Amount of Sample required 6. Sample handling and preservation 7. Equipment required 8. Reagents Required 9. Procedure	**Training requirements** - WATS has a written SOP for training personnel to perform test methods, as well as documentation of that training. This SOP has been attached to this response letter, and will also be included as an attachment to the Multi-Area QAPP. **Health and safety** - In addition to the health and safety topics covered within the test methods and SOPs, WATS has a Chemical Hygiene Plan explaining an employee's right to know with regard to working with hazardous chemicals and the medical care that an employee has the right to receive. Training with regard to the chemical hygiene plan is given during the employee's first week of employment with annual refreshers. This chemical hygiene plan is available to the USEPA upon request.
Quality Assurance Quality Control (QA/QC)	Discuss appropriate QA/QC procedures	Quality Assurance/Quality Control procedures are included as Section 10.	No modifications to meet objective.
References	List any significant references	References are included as Section 12.	No modifications to meet objective.

1.0 SCOPE

1.1 This document specifies the duties and responsibilities of the Sample Management Office (SMO) and those of the employees coming into contact with the samples.

1.2 SMO plays a critical role in the Unit's QA program through receipt, handling, documentation, and disposal of samples for Analysis and Testing.

1.3 This SOP only applies to samples submitted to the pulp (wet) lab, paper lab, and to the analytical laboratories. Microscopy has a separate procedure for the sample management process. See MP-SAMPLE RECEIVING.

2.0 SAMPLE SUBMITTAL PROCESS

The following forms are found on the A&T website under "Forms & Templates" in the SMO folder:

- Sample Receipt Form
- Sample Analysis Request & Chain of Custody Record
- Analytical Chemistry Service Request
- Pulp & Paper Service Request
- Microstructure Service Request

To submit a sample, the following forms are found on the A&T website under "Submit a Sample" to request the sample testing schedules:

- Analytical Chemistry Service Request Form
- Pulp & Paper Service Request
- Microstructure Service Request

See section 4.0 below for more information regarding how to locate the necessary forms.

3.0 REAGENTS REQUIRED

3.1 HNO_3 (1+1) with water. Add 250 mL of conc. acid to 250 mL of water and mix. Need not be extremely accurate.

NOTE: For safety and purity reasons, this should be made up by metals laboratory personnel, not SMO personnel.

Nitric acid (concentrated), HNO_3, CAS# 7697-37-2

DANGER: Handle with extreme care. Wear safety glasses, lab coat, vinyl gloves and use in a hood. Conc. nitric acid is a strong, corrosive acid and liquid and vapor can cause severe burns. Carefully add acid to water with frequent or continuous stirring. Mix in a tub or stoppered sink. Upon dilution in water, the generation of heat could cause it to erupt and spatter over a large area. Proceed with caution.

- Harmful if inhaled and may cause delayed lung injury. If inhaled, can cause severe irritation or burns of mucous membranes and respiratory system, resulting in coughing, difficult breathing, chest pains, pneumonia, pulmonary edema, lung inflammation, unconsciousness, and may be fatal. Can cause severe ulceration.
- Skin contact can cause severe irritation, redness, pain, and severe skin burns. Concentrated acid causes deep ulcers and stain skin a yellow or yellow-brown color.
- Eye contact can cause severe irritation or burns and result in permanent damage.
- Nitric acid is a strong oxidizer. Contact with combustible materials, flammable materials - such as wood and solvents - or powdered metals can cause fire or explosion. Reacts with most metals to produce hydrogen gas, which can form an explosive mixture with air. Keep away from strong bases, carbides, carbonates, charcoal, (hydrogen) sulfides, cyanides, combustible materials, combustible organics or organic materials, turpentine, strong reducing agents, most common metals, carbides, ammonium hydroxide, water, and alcohols.
- Certain mixtures with benzene, 1,2-dichloroethane, or dichloromethane may be detonatable.
- Reaction products include highly toxic and dangerous fumes of reddish oxides of nitrogen having the equivalent affects as those noted above.
- Long-term exposure to concentrated vapors may cause erosion of teeth.
- Short-term exposure limit (STEL) is 10 mg/m^3 (4 ppm) and permissible exposure limit (PEL) is 5 mg/m^3 (2 ppm.)

4.0 PROCEDURE

SMO is responsible for the general cleanliness of the sample storage and receiving areas. A clean and orderly storage area is necessary for the integrity of the samples. Important records can be lost if paperwork is not kept organized.

4.1 Location of forms to submit a sample

Go to A&T web site

Click on "Submit a form". Under Service Request forms, choose from the following: Pulp and Paper, Microstructure, Sample Analysis & Testing Request with a chain of Custody form or Sample Analysis & Testing Request without a chain of custody.

4.2 Receiving Samples

Sample Management Office (SMO) receives samples through a variety of methods.

4.2.1 Samples are brought directly into SMO by the client. If no one is present in SMO, the client should consult with one of the Project Managers (PM) about his sample request.

4.2.2 Samples are shipped and/or mailed to the Technology Center via various methods.

a. Samples are accepted by our receiving personnel and delivered to the SMO. (At times, SMO will come down and retrieve samples from the receiving dock.)

b. On occasion, samples will be dropped off in the south lobby area. The receptionist will then contact SMO to pick-up the samples.

NOTE: Although section 4.1.2.b is still listed as a viable method for sample drop off, it is strongly discouraged due to safety reasons.

4.2.3 Samples are brought in to SMO by the analyst.

a. Samples are sent or given directly to the analyst by the client.

b. On occasion, samples are created by an analyst in collaboration with a client.

4.2.4 Samples are delivered to the Technology Center during off-hours.

Security handles samples as per their SOP.

a. Samples received in coolers are placed in the basement cold storage G01/G02 and security sends a voice mail message to the SMO personnel.

b. Samples not received in coolers (not to be kept chilled) are placed in the receiving area and delivered to SMO at the start of the following business day.

4.3 Project Manager Role

When samples arrive, the SMO staff calls a Project Manager (PM). The PM is determined by the client/job type received.

4.4 Unpack and evaluate the samples.

The following is a list of procedures performed in the sample check-in process. See AQ S-SampleSplitting if needed. (Section 4.4.1.j)

4.4.1 Sample handling.

THE INTEGRITY OF ALL SAMPLES SHALL BE MAINTAINED WHILE IN LABORATORY CUSTODY. FOR EXAMPLE, IF SAMPLES REQUIRE 4°C, THEY WILL BE MAINTAINED AT 4°C DURING ANY HANDLING PROCESSES (except when warming them for sub-sampling).

SAMPLES WILL BE APPROPRIATELY PROTECTED FROM CROSS-CONTAMINATION. FOR EXAMPLE, THE VOAs OR HI-RES SAMPLES WILL BE ISOLATED TO KEEP FROM POTENTIALLY CONTAMINATING OTHER SAMPLES.

a. For samples received chilled or in coolers, the SMO staff uses the Sample Receipt Form found on the A&T website under "Forms & Templates" in the SMO folder. This form is used to record, date and time samples are received, pH conditions, temperature of cooler/temperature blank and custody seal conditions, etc.

b. When temperatures are in question (i.e. no ice present or blue ice totally melted), a probe is used to check temperatures. The probes are stored in the cabinet under sample fume hood 216-4.

If there are no coolant blanks, place the probe in the cooler next to a sample bottles for the most accurate ambient temperature. Make a note on the sample receipt form how the temperatures were measured (coolant blank, water, ambient air).

c. Unpack samples and check for breakage, missing samples and correct identification, etc.

NOTE: TOTALLY UNKNOWN SAMPLES, OR SAMPLES KNOWN OR SUSPECTED TO BE HAZARDOUS, ARE TO BE OPENED AND INSPECTED IN A LABORATORY HOOD ONLY

Broken sample containers, irregular preservation and improper paperwork are noted on a nonconformance form as per OQ CORRACT. The assigned PM is contacted so a course of action can be taken such as the following:

- Contacting clients so more samples can be submitted
- Reducing the number of tests requested by the clients
- Contacting clients regarding future packing of samples

If anything is unclear, special or requires immediate handling about a client's request, the SMO personnel will contact the assigned PM or the appropriate analysts for assistance.

d. The SMO personnel (who unload coolers, boxes, etc.) will verify that samples and accompanying paperwork match. The SMO personnel will promptly sign all of the chain of custody forms.

When there is no client paperwork, the SMO personnel contact the project managers to see if they have any knowledge of the work that was received. When project managers have no knowledge of samples received, the SMO personnel will then look on the package for sender information (i.e. mill, company or name on the package) so that the SMO personnel or project managers can contact the sender to find out what kind of analyses are required.

All paperwork that accompanies the incoming samples is kept together and becomes part of the original SR package.

e. Determine if the samples received are within holding times.

Holding Time	Action
No Issues	Process normally
Exceeded	Contact PM, laboratories affected and complete a nonconformance
Short	Contact PM and laboratories affected immediately

f. Determine if the samples are properly preserved.

1. Using the Sample Receipt form, confirm which samples have a pH preservation requirement and require a pH measurement to be taken. Retrieve the pH test strips (these are located in both hoods ((216-3 & 216-4)) in the SMO area.

i. To take a pH of a sample, pour a small amount of the sample into a disposable 50-mL polypropylene beaker. Dip the pH strip into the liquid.

ii. Pour excess sample down the sink with the water running and the dip sticks in the trash.

2. Most samples needing their pH checked should be preserved to a pH < 2. Samples requiring more H_2SO_4, or HNO_3 (if not designated as low level metals), may be preserved by SMO personnel or taken to the lab for preservation.

CAUTION: Be aware that an unpreserved bottle (for metals analysis) may be for silicon testing and does not require preservation. See chain of custody and/or other testing information before adding any preservatives to such sample containers.

DANGER: Handle with extreme care. Nitric acid is a strong, corrosive mineral acid. Use all appropriate safety apparel and proceed with caution. See 'DANGER' under 3.1.

3. Cyanide pH should be > 12. If the cyanide sample has not been preserved and appears to have an improper pH, take the sample to the Lab 212 SLM and have a chemist preserve it to a pH >12 by adding 2 mL of 10N NaOH per liter of sample.

4. Use lead acetate strips to check for presence of sulfide in liquid samples where cyanide is requested. If sulfide is present in the sample, it will be indicated by a darkening of the test strip or a dark precipitate on the test strip due to PbS (lead sulfide) precipitating out with color (black). Record pH and sulfide information on Sample Receipt Form (Section 4.4.1.a).

5. Check other preservation required as specified, such as RCl_2 (residual chlorine), as necessary for N. Carolina regulatory samples.

g. Add any needed preservative in accordance with proper analytical protocol as stated in 40 CFR 136.3, Table II. See a copy of Table II in the appendix of this document.

h. Preservation is done immediately upon receipt in the lab and recorded on Sample Receipt Form.

i. If initial attempts to add preservative do not achieve the desired effect, contact an analyst to see about adding a more concentrated preservative. Total volume of preservative should not dilute the sample by more than 1 % (10 mL/L).

j. Evaluate whether there is sufficient sample to run the requested tests. If there is not, or if there is doubt, consult AQ S-SampleSplitting and/or contact the PM. Should testing proceed when there is insufficient sample, a nonconformance memo will be filled out.

k. If the samples are in an unusual or abnormal state, such as lack of proper preservative for testing (i.e. H_2SO_4 rather than HNO_3), the PM is notified and a determination is made on how to proceed. This may require a note on the sample log-in sheet under "*Comments*" or a nonconformance form may need to be completed.

4.4.2 Determine if paperwork matches samples. If not, then the PM is informed and a nonconformance memo will be filled out.

4.4 3 Evaluating Service Request (SR) needs:

a. Determine if an 'upcoming request' was created. Look in the "UPCOMING" notebook for copy.

If:	
Yes	Contact appropriate PM that samples have arrived. Proceed with log-in following detail on upcoming.
No	Contact appropriate PM for analysis details. These details would include: •who the client is •client location and phone number •correct project number for charging •correct tests and corresponding test codes •special instructions regarding these samples •reference SR number •confirmation of a committed turnaround time •lab doing the work

b. When there is an upcoming notice for a particular job and the samples that are received do not match the initial notice (i.e., expect 41 samples, receive only 7 in current shipment), make a copy of the upcoming notice. This copy goes with the new SR being created. Write on the original copy of the upcoming notice the number of the SR created for the work received and place the upcoming notice back into the notebook for future samples to be received for that same job.

c. Determine if there is a reference Service Request (SR).

1. A reference SR number is noted on the upcoming notice by the PM if we've done previous work for that client/project.

2. If a referenced SR number is not noted on the upcoming notice, open 'A&T SR Search' from the main screen of Laboratory Information System (LIMS).

3. A LIMS search is based on several criteria.
   i. Mill site, such as "Columbus".
   ii. Client, such as "Mike White".

4. The search is much quicker using only the upper part of the LIMS page. (Most searches will give the information required when using the upper part of the page.) Entering a test or test schedule (shown on the lower half of the page) will be much broader and take longer.

4.4.4 When the list of SRs comes up on the LIMS screen, the most recent SR is at the top (this is not true when searching the committed database). If the information being searched for is not found in the active database, try searching under the committed database. Active/committed SR's can be opened and printed by clicking on the database header in LIMS & toggling between 'Toggle act/com'.

NOTE: SRs are moved into the committed database after an SR has been closed for 120 days.

4.5 Enter sample/requested information/documentation into LIMS

After all questions are answered and issues are resolved, the SMO personnel are ready to enter the information into the Laboratory Information Management System (LIMS) and create a unique Service Request (SR) for that particular sample set. This document (SOP) does not cover the detail of sample entry into the LIMS. See LIMS notebook located in the SMO area for LIMS entry instruction.

NOTE: SR numbers are assigned in sequential order by the computer. The SR number is a 6 digit number, with the first 2 digits designating the year, e.g., 2007 becomes 07-, and the last 4 for the sequential SR in that year, allowing up to 9999 SR's in a given year. The 4985th SR of 2007 would be 07-4985. See section 4.5.2 for labeling information.

If a sample is deleted, the LIMS does not allow that particular ID number to be reused. Not all sample ID's will be sequential

4 5 1 After all information is entered into the LIMS and the SR is complete, print one full SR copy. Print one single copy of the front page of the SR and mark F in upper right corner. The F copy is placed in the SR file at the back of the SR and accompanying paperwork for archival filing after reporting. Scan the client paperwork into LIMS. Place all paperwork for SR in the appropriate folder.

Folder Color	Folder Contents
Green	'Permit' in title
Red	"outside" SRs
Yellow	Analytical

NOTE: No folder is made for the Cellulose Properties samples. All work is scanned into an electronic copy However, if the Cellulose Properties SR has one or more tests for Analytical Chemistry, a yellow folder will be made and stored as in 4.5.1

Place file folder in rack next to SR printer. Notify appropriate PM electronically when SR's are ready for reviewing.

4.5.2 The PM will come to SMO and pick up SR folders. The PM will review the SR's either in SMO or at their desk to evaluate whether all aspects of the SR are in agreement with both the laboratory and the client. If no changes are required, then the PM 'releases' the SR to the laboratories (with scanned attachments) via a shared e-mail file This puts the SR in the LIMS system so the laboratories are able to enter their data.

4.5.3 IR LAB: SMO will print a full copy of the SR, scan the client paperwork and create a folder. This folder is reviewed by a SMO technician who did not enter the SR. After review, corrections or changes, the SR is sent electronically to the IR specialist for review and released to the labs. The SR folder is placed behind the purple folder labeled Mary Beth at the back of the SR rack which is located next to the scanner. The IR specialist will either pick up the SR folder to place in the SR file cabinet drawer or SMO personnel will take it out to the drawer.

4.5.4 When the PM 'releases' the SR, it is seen in the exchange file. The SR number shows up followed by numbers. Each number represents an area of the lab. Some areas are broken down further by specialty and these show up with a letter, i.e. 4p indicates group 4 or Conventionals and the pulp area of that lab. The list below shows the number of each lab and it is the lab number that is used when sending the SR electronically. When a group opens the exchange file, they need only look for SRs with their own group number attached. Remember that all attachments are with the lab copy.

0 Project Management/QC
1 Chromatography
2 IR/Voa/Dioxin, with i (IR), v (VOA) or d (dioxin)
3 Elemental
4 Conventional, with *w* (waters) or *p (pulp)*
6 Physical Test Lab (Paper Lab)
7 Pulp Lab (Wet Lab)
8 Containerboard Lab
9 Microscopy

Changes can be made and/or samples can be added to an SR until it is authorized (final closing).

NOTE: Archiving of completed SRs is a function of the Administrative personnel.

Exemptions

MICROSTRUCTURE:

SMO will print a full copy of the SR and scan the client paperwork as directed above. This SR is reviewed by a SMO technician who did not enter the SR. After review, corrections or changes, the SR is sent electronically to microstructure for review and released to the labs. The complete SR is put in WOW box (micro will print their own copies). The client paperwork goes in a purple folder labeled Ron which is at the back of the SR rack next to the SR scanner. The micro PM either prints this paperwork at his desk, while SMO retains the original paperwork until the file folder gets too full. At that time, SMO will discard the oldest sets of paperwork and continue to rotate paperwork in and out of that folder. Samples for Microstructure are usually delivered directly to that lab area. The procedure follows MP-SAMPLE RECEIVING.

CFTP (formerly PPTS):

SMO will print a full copy of the SR and scan the client paperwork as directed above. The SR is reviewed by a SMO technician who did not enter the SR. After review, corrections or changes, the SR is sent electronically to CFTP OM/PM for review and released to the labs. The complete SR is put in WOW box (CFTP will print their own copies). The client paperwork goes in a purple folder labeled Rick which is at the back of the SR rack next to the scanner. The CFTP OM/PM also prints this paperwork at his desk, while SMO retains the original paperwork until the file folder gets too full. At that time, SMO will discard the oldest sets of paperwork and continue to rotate paperwork in and out of that folder.

4.7 Label Samples

4 7.1 Each sample is assigned a unique lab code number. This is done through the process of logging the samples into the computer on a given SR. The lab code numbers are assigned sequentially by the computer. When new samples are "created" (such as by combining samples together), lab code numbers will be created for these as well. An SR can contain up to 999 samples.

NOTE: Generally, for work internal to the company, containers known to hold split samples from one identical source, are given the same number, being differentiated by bottle type/preservation.

Each sample number contains the 6 digit SR number, which is the two digit year, then a "–" (dash) followed by the next sequential SR number, another dash, and the first sample number, starting with

001. Each additional sample is assigned the next sequential number (e.g. 002,003, etc.). See NOTE in section 4.6.1 for more information.

Example: 00-1925-001, 00-1925-002, etc

4.7.3 Print sample labels. This is done through the LIMS. If available from the client, the label also contains the sample date and time.

4.7.4 Attach labels to each sample container. When affixing the sample number label, take care not to cover the sample detail information on the sample container such as sample ID, date/time/preservative. Also take care not to damage samples with the label in cases where there is no container, such as with pulp sheets. In this case, attach the sample label by using a paper clip or stapling.

NOTE: See SMO staff for when to staple and when to use paper clips. Frequently, samples for the Wet Lab are taken directly to the Wet Lab by the client. (See section 4.8.12.)

4.7 5 All pulp samples going to the Paper Laboratories or to the 20 % room need to have sample labels paper clipped directly to the samples. DO NOT USE STAPLES. Ensure that sample label backing is intact so that nothing "sticky" adheres to the sample. For pulp samples requiring metals analysis, wear cotton gloves while handling samples, place samples in plastic bags and ensure no metal is touching the pulps. For pulp samples requiring Conventionals testing, wear cotton gloves while handling samples, place samples in plastic bag. For IR samples, place in envelopes. Do not staple, put in plastic or use paperclips.

4 8 **Storing Samples**

4.8.1 SMO personnel stores samples appropriately to maintain the integrity of the samples and the safety of the lab. Samples generating hazardous fumes require placement in a hood. Samples that will deteriorate are refrigerated or frozen. Light sensitive samples are kept in the dark.

4.8.2 There is one 65 m^3 (2,300 ft^3) walk-in cooler in the SMO area. Refrigerated samples go directly to this location after initial check-in. This refrigeration unit is large enough to accommodate a large number of sample containers. There is one 0.6 m^3 freezer (located in lab 227 SLM) for frozen samples. Non-perishable samples are kept in cupboards, shelves, and drawers in the SMO area.

4.8.3 After the Service Request is generated, label the samples and assign a storage location. (See section 4.7 - Label Samples.)

4.8 4 For each SR, complete a Sample Log Sheet. This sheet is placed in the Sample Log Book for samples processed through the SMO. There is one book for Analytical Chemistry and another for the Wet Lab, Paper Lab, & Microscopy labs. The SR is created by SMO for Microstructure, but samples do not usually come through the SMO. The Sample Log Sheet page is designed to track the location and status of samples that come into the lab. For Analytical Chemistry, each SR is assigned a separate page. Enter the SR number, the initials of the person handling the samples, the date you are placing samples in the location, the inclusive sample numbers, number of containers (bottles and bottle type (i e.- 2X1L MET), plastic bag, envelope, etc.) and the location to which the samples are assigned.

4.8.5 For Wet Lab, Paper Lab, & Microstructure (when necessary), a modified sheet in a separate book is used in the SMO. One SR per line is used. In addition to the SR, enter the initials of the person

who placed the samples in a given location, the inclusive sample numbers, the date samples are processed, and the location to which the samples are assigned.

4.8.6 For most of the routine work done for Analytical Chemistry, samples will be assigned to storage locations in the SMO and the laboratory personnel will check out samples from the SMO area. Preserved samples (in particular, those for metals) that can be stored at room temperature should be placed in cupboards C1 or C2. Liquors, caustics, acids, and samples other than waters that are held at room temperature are placed in C3. Secondary containment (plastic 'tubs') is used for all liquors, caustics, and acids. Care should be taken to store samples from each service request together and not to mix samples from more than one SR in the secondary containment 'tub.'

WARNING: Do not store caustics and acids from the same SR together in the same tub. Accidental spills may react violently and burn or otherwise injure personnel.

4.8.7 Use the following codes to designate location in SMO:

216V: where 216 = SMO walk-in cooler and V = assigned shelf number.
D-#: where D = drawer and # =drawer number.
C-#-S: where C = cabinet, # = cabinet number, and S = shelf letter.
S-#. where S = shelf and # = shelf number.

4.8.8 EXEMPTION: VOA lab. Place all samples for volatiles analysis directly in the refrigerator located in the volatiles lab (223 SLM). This location is noted as the lab number then V-1 (223-V-1). Round Robin samples (usually small ampoules) are placed upright in a small beaker and stored in the VOA lab freezer (223 F-1).

4.8.9 When samples are picked up by the lab before being assigned a SMO location, they are noted as lab number followed by P/U (pick-up). If lab 212 SLM picks up samples directly from SMO the location is noted as 212 P/U which indicates that samples were picked up by someone from lab 212. This could be used by any lab location. On occasion, samples may be assigned to other locations. Apply an appropriate description, e.g., "northeast hood in 211," "Eatherton-233," or "G01-'S#.'" Use 'R' when designating refrigerators in lab rooms, e.g., R226N for the refrigerator in NLM 226, and 'F' for freezers. For large projects, samples may need to be kept in the cold room downstairs (G01 or G02).

4 8.10 Analysts shall sign samples out when taking them and sign them back in when they are returned. The log also contains a brief description of any sample modification or preparation, including any sub-sampling.

a. Employees taking samples will log the sample location (not the lab that the employee works in) to which the sample will be taken. At times, more than one lab will be working on the same sample and oral communication takes place regarding the current location of the sample. At the end of the shift, one of these employees logs the sample's location in the Sample Log Book sheet.

b. In the case of sample extractions requiring further analytical work, the extractives will be logged on the Sample Log sheet as to the location in SMO and with the appropriate comments as to the sample preparation performed. For example, a TCLP sample may be prepared in the Metals Lab. This sample will require further analysis in the Chromatography lab. The Metals personnel will return the TCLP sample to SMO, log its location and notify the Chromatography lab of the samples readiness and location. If the Metals personnel puts the

samples in the Chromatography lab in order to be helpful, the Metals personnel must still log the location of the sample in the SMO Sample Log sheet.

4.8.11 Samples that are completely used during analysis, (except for O & G), or are returned to the client by analysts, should be noted on the sample log sheet by that analyst with the appropriate 'Disposal' and 'Comment' code noted at the bottom of the Sample Log sheet.

4.8.12 Samples for the Wet Lab are taken to Lab 105 in the high bay. Most are placed in the 50%- RH conditioning room, 105C. Room 105C has boxes and shelves noted for sample drop off. There is a clipboard in 105C where the SMO staff records the delivered samples. The information on the clipboard includes the SR#, the location of the samples and the date that the samples are delivered to the lab. Wet samples are stored in the lab refrigerator, located in lab 105 and are noted on the clipboard sheet.

4.8.13 Most samples for the Paper Lab are placed by the paper lab personnel in the 20% conditioning room overnight. The 20% conditioning room is at the north end of the west corridor on the second floor. There is a clipboard in the 20 % conditioning room where the storage location is noted. The information on the clipboard includes the SR#, the shelf location, and the date samples were delivered to that room. If samples are clipped to wire, the location is wire. Samples for the Paper Lab are picked up daily by paper lab personnel and taken to the appropriate locations.

4.8.14 If the sample for Paper Lab requires optical testing (opacity, scattering, brightness) and is a light-sensitive product, such as newsprint, the sample should be placed (if not so received) in black plastic to prevent discoloring.

4.9 **Disposing of Samples**

4.9 1 Unless otherwise requested, the SMO holds samples that have been assigned to Analytical Chemistry for one month after closing an SR. Samples assigned to Wet Lab or Paper Lab are disposed of or returned to the client by the staff in those areas and never returned to the SMO, unless requested.

4.9.2 Final disposition could be sewering water samples, returning sample to the client, or passing appropriate samples to the Chemical Management unit.

4.9.3 Properly dispose of samples as per the disposal procedure in the appendix of the Chemical Hygiene Plan.

NOTE: If unsure of sample type or disposal method needed, see Chemical Hygiene Officer.

4.9.4 If samples were returned to the client, the return is entered into LIMS. Click on SR+Samples, then click on enter disposal to SR. Type in SR number that needs to be returned. Where it says "actual disposal", choose R for return, where it says "disposal date", enter the date that the sample was returned it to the client.

4.9 5 Upon completion of work, the sample log page is removed, updated as needed and filed in a separate notebook in numerical order. These records will be treated in the same manner as Archived Service Requests (ASR) for storage purposes. (See OQ DOCTRL.)

5.0 QUALITY ASSURANCE

5.1 The refrigerators and freezers are checked daily by assigned personnel from the lab per AQ O-DLYCHK. Refrigerators are kept in the range of 4 ± 2 °C and freezers at <-15 °C. If abnormal conditions are discovered, they are noted in the temperature logbook and the SMO is notified. SMO then takes appropriate action to correct the situation. This may include replacing a thermometer, notifying the project manager, calling (x4200) to initiate repairs, and/or transferring samples to other locations. Lab personnel also make notice of unusual situations in their daily use of the refrigerators.

5.2 Nonconformances include, but are not limited to:

- Broken sample containers
- Irregular preservation or lack of proper preservative
- No COC (Chain of Custody) or improper paperwork
- Insufficient sample
- Received sample temperatures warm
- Id's don't match customer lists

6.0 KEY WORDS

OP S-SMO, OP S-SAMPSPLIT, sample disposal, Sample Receipt Form, Sample Log sheet, sample submission, sample management, sample management office, SMO, Chain of Custody (COC), Analytical Chemistry Service Request Form, Pulp & Paper Service Request Form

7.0 REVISION HISTORY

7.1 05 SEPT 2006: Complete revision and update of SMO processes.

7.2 01 FEB 2007: Updated document to reflect changes in Weyerhaeuser structure – PPTS is now known as CFTP.

7.3 28 FEB 2007: Added safety note to section 4.1.2, added information to section 4.4.1.d, changed SOP identification.

7.4 13 JUL 2007: Added references to the appropriate departmental SOPs, corrected typo and numbering errors, corrected section 4.5.1 and added sections a and b to 4.8.10. Also added section 4.8.11 and the appendix. Deleted Table of Contents.

Appendix

40 CFR136.3

TABLE II—REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES

Parameter No./name	Container[5]	Preservation[2,3]	Maximum holding time[4]
Table IA—Bacteria Tests:			
1–4. Coliform, fecal and total	P,G	Cool, 4C, 0.008% $Na_2S_2O_3$[6]	6 hours.
5. Fecal streptococci	P,G	Cool, 4C, 0.008% $Na_2S_2O_3$[6]	6 hours.
Table IA—Aquatic Toxicity Tests:			
6–10. Toxicity, acute and chronic	P,G	Cool, 4 °C[16]	36 hours.
Table IB—Inorganic Tests:			
1. Acidity	P, G	Cool, 4°C	14 days.
2. Alkalinity	P, G	do	Do.
4. Ammonia	P, G	Cool, 4°C, H_2SO_4 to pH<2	28 days.
5. Biochemical oxygen demand	P, G	Cool, 4°C	48 hours.
10. Boron	P, FFTE, or Quartz.	HNO_3 TO pH<2	6 months
11. Bromide	P, G	None required	28 days.
14. Biochemical oxygen demand, carbonaceous	P, G	Cool, 4°C	48 hours.
15. Chemical oxygen demand	P, G	Cool, 4°C, H_2SO_4 to pH<2	28 days.
16. Chloride	P, G	None required	Do.
17. Chlorine, total residual	P, G	do	Analyze immediately
21. Color	P, G	Cool, 4°C	48 hours.
23–24. Cyanide, total and amenable to chlorination.	P, G	Cool, 4°C, NaOH to pH>12, 0.6g ascorbic acid[9].	14 days.[8]
25. Fluoride	P	None required	28 days.
27. Hardness	P, G	HNO_3 to pH<2, H_2SO_4 to pH<2	6 months.
28. Hydrogen ion (pH)	P, G	None required	Analyze immediately.
31, 43. Kjeldahl and organic nitrogen	P, G	Cool, 4°C, H_2SO_4 to pH<2	28 days.
Metals:[7]			
18. Chromium VI[7]	P, G	Cool, 4 °C	24 hours.
35. Mercury[17]	P, G	HNO_3 to pH<2	28 days.
3, 5–6, 12,13, 19, 20, 22, 26, 29, 30, 32–34, 36, 37, 45, 47, 51, 52, 56–60, 62, 63, 70–72, 74, 75. Metals except boron, chromium VI and mercury[7].	P, G	co	6 months.
38. Nitrate	P, G	Cool, 4°C	48 hours.
39. Nitrate-nitrite	P, G	Cool, 4°C, H_2SO_4 to pH<2	28 days.
40. Nitrite	P, G	Cool, 4°C	48 hours.
41. Oil and grease	G	Cool to 4°C, HCl or H_2SO_4 to pH<2.	28 days.
42. Organic Carbon	P, G	Cool to 4 °C HCl or H_2SO_4 or H_3PO_4, to pH<2.	28 days.
44. Orthophosphate	P, G	Filter immediately, Cool, 4°C	48 hours.
46. Oxygen, Dissolved Probe	G Bottle and top.	None required	Analyze immediately.
47. Winkler	do	Fix on site and store in dark	8 hours.
48. Phenols	G only	Cool, 4°C, H_2SO_4 to pH<2	28 days.
49. Phosphorus (elemental)	G	Cool, 4°C	48 hours.
50. Phosphorus, total	P, G	Cool, 4°C, H_2SO_4 to pH<2	28 days.
53. Residue, total	P, G	Cool, 4°C	7 days.
54. Residue, Filterable	P, G	do	7 days.
55. Residue, Nonfilterable (TSS)	P, G	do	7 days.
56. Residue, Settleable	P, G	do	48 hours.
57. Residue, volatile	P, G	do	7 days.
61. Silica	P, FFTE, or Quartz.	Cool, 4 °C	28 days.
64. Specific conductance	P, G	do	Do.
65. Sulfate	P, G	do	Do.
66. Sulfide	P, G	Cool, 4°C add zinc acetate plus sodium hydroxide to pH>9	7 days.
67. Sulfite	P, G	None required	Analyze immediately.
68. Surfactants	P,G	Cool, 4°C	48 hours.
69. Temperature	P, G	None required	Analyze.
73. Turbidity	P, G	Cool, 4°C	48 hours.
Table IC—Organic Tests[8]			
13, 16–20, 22, 24–25, 34–37, 39–43, 45–47, 66, 76, 104, 105, 108–111, 113. Purgeable Halocarbons.	G, Teflon-lined septum.	Cool, 4 °C, 0.008% $Na_2S_2O_3$[6].	14 days.

TABLE II—REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES—Continued

Parameter No./name	Container[1]	Preservation[2,3]	Maximum holding time[4]
6, 57, 106. Purgeable aromatic hydrocarbons	___co	Cool, 4 °C, 0.008% $Na_2S_2O_3$,[5] HCl to pH2[9].	Do.
3, 4. Acrolein and acrylonitrile	___do	Cool, 4 °C, 0.008% $Na_2S_2O_3$,[5] adjust pH to 4–5[10].	Do.
23, 33, 44, 49, 53, 77, 80, 91, 95, 100, 112. Phenols[11].	G, Teflon-lined cap.	Cool, 4 °C, 0.008% $Na_2S_2O_3$[6]	7 days until extraction; 40 days after extraction
7, 38. Benzidines[11]	___co	___do	7 days until extraction.[12]
14, 17, 48, 50–52. Phthalate esters[11]	___co	Cool, 4 °C	7 days until extraction; 40 days after extraction.
22–84. Nitrosamines[11,14]	___co	Cool, 4 °C, 0.008% $Na_2S_2O_3$,[5] store in dark.	Do.
23–54. PCBs[11]	___co	Cool, 4 °C	Do.
54, 55, 75, 79. Nitroaromatics and isophorone[11]	___co	Cool, 4 °C, 0.008% $Na_2S_2O_3$,[5] store in dark.	Do.
1, 2, 5, 8–12, 32, 33, 55, 56, 74, 75, 99, 101. Polynuclear aromatic hydrocarbons[11].	___do	___do	Do.
15, 16, 24, 31, 67. Haloethers[11]	___co	Cool, 4 °C, 0.008% $Na_2S_2O_3$[6]	Do.
25, 35–37, 63–65, 73, 107. Chlorinated hydrocarbons[11].	___co	Cool, 4 °C	Do.
60–62, 66–72, 85, 86, 96–97, 102, 103. CDDs/CDFs[11].			
aqueous: field and lab preservation.	G	Cool, 0–4 °C, pH<9, 0.008% $Na_2S_2O_3$.[5]	1 year.
Solids, mixed phase, and tissue: field preservation.	___co	Cool, <4 °C	7 days.
Solids, mixed phase, and tissue: lab preservation	___co	Freeze, <= 10 °C	1 year.
Table ID—Pesticides Tests:			
1–70. Pesticides[11]	___co	Cool, 4°C, pH 5–9[13]	Do.
Table IE—Radiological Tests:			
1–5. Alpha, beta and radium	P, G	HNO_3 to pH<2	6 months.

Table II Notes

[1] Polyethylene (P) or glass (G). For microbiology, plastic sample containers must be made of sterilizable materials (polypropylene or other autoclavable plastic).

[2] Sample preservation should be performed immediately upon sample collection. For composite chemical samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.

[3] When any sample is to be shipped by common carrier or sent through the United States Mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of Table II, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

[4] Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that for the specific types of samples under study, the analytes are stable for the longer time, and has received a variance from the Regional Administrator under § 136.3(e). Some samples may not be stable for the maximum time period given in the table. A permittee, or monitoring laboratory, is obligated to hold the sample for a shorter time if knowledge exists to show that this is necessary to maintain sample stability. See § 136.3(e) for details. The term "analyze immediately" usually means within 15 minutes or less of sample collection.

[5] Should only be used in the presence of residual chlorine.

[6] Maximum holding time is 24 hours when sulfide is present. Optionally all samples may be tested with lead acetate paper before pH adjustments in order to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.

[7] Samples should be filtered immediately on-site before adding preservative for dissolved metals.

[8] Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.

[9] Sample receiving no pH adjustment must be analyzed within seven days of sampling.

[10] The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.

[11] When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6–9; samples preserved in this manner may be held for seven days before extraction and for forty days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (re the requirement for thiosulfate reduction of residual chlorine), and footnotes 12, 13 (re the analysis of benzidine).

[12] If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0±0.2 to prevent rearrangement to benzidine.

[13] Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.

[14] For the analysis of diphenylnitrosamine, add 0.008% $Na_2S_2O_3$ and adjust pH to 7–10 with NaOH within 24 hours of sampling.

[15] The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% $Na_2S_2O_3$.

1.0 SCOPE

This method is applicable to the determination of suspended solids in pulp and paper effluents, wood products effluents, raw and treated water and natural waters.

Suspended solids are defined as those solids retained by a standard glass fiber filter.

This method is based upon EPA method 160.2. It replaces method AM1-160.2.

2.0 SUMMARY OF METHOD

A well-mixed sample is filtered through a standard glass fiber filter and the residue retained on the filter is dried to a constant weight at 104 ± 1°C.

3.0 INTERFERENCES

Non homogeneous materials, such as leaves, sticks, and large floating particles, should be excluded from the sample.

Some solids will decompose below the method required temperatures.

Biological organisms, such as algae and insects, are generally not considered to be part of the solids. Problems associated with these should be noted on the report, especially for regulatory monitoring. In some cases, method modifications may be needed to generate an estimate of the suspended solids.

Too much residue on the filter will entrap water and may require prolonged drying times. Clogging of the filter with residue will also prolong filtering time and retain small particles which would normally pass through the filter.

4.0 ESTIMATE OF ANALYTICAL TIME

An average sample will take 10 to 15 min to run. 10 normal samples may be run in 30 to 40 min. Samples with plugging, or high solids problems may take considerably longer, depending upon the nature of the sample.

5.0 SAMPLE SIZE REQUIRED

There is no fixed requirement for sample volume. Normal samples will take about 100 mL. Samples higher in solids may need considerably less. Samples very low in solids may require up to a liter or

more; however, greater volumes decrease the accuracy of the test by increasing the amount of materials being washed from the filter. Normally volumes over 500 mL are not recommended. Include single sample, plus amount needed for QC. This may include a desired amount, in case of need for re-running, or minimum.

6.0 SAMPLING, SAMPLE HANDLING, AND PRESERVATION

Samples should be collected in clean polyethylene or glass containers. Samples should be analyzed as soon as possible after collection, but can be held under refrigeration at < 4°C for seven days, if immediate analysis cannot be performed.

7.0 EQUIPMENT REQUIRED

7.1 Gelman membrane-style filter holder & membrane filter funnel.

7.2 4.7-cm glass fiber filter discs, without organic binder, Gelman type A/E.

7 3 Vacuum source.

7.4 Drying Oven.

7.5 Aluminum pans. (50-mm diameter)

7.6 Graduated cylinders, TC (to-contain) class B

7.7 Desiccator (with Drierite desiccant.)

7.8 Analytical balance capable of weighing to 0.1 mg.

7.9 Vacuum flask, 500-mL or 1-L.

8.0 PROCEDURE

8.1 Preparation of glass fiber filter disc: Place the disc on the membrane filter apparatus. Apply vacuum and wash the disc with three successive 20 ± 2-mL volumes of water. This is to remove small loose fibers and wash away wet strength material from the filter. Allow each washing to completely pass through the filter before beginning the next. Continue suction to remove traces of water from filter disc. Release the vacuum. Remove filter disc from apparatus and transfer to a numbered aluminum pan.

8.2 Dry aluminum dish and glass fiber filter for at least one hour at 104 ± 1°C.

WARNING: Skin contact with hot surfaces may burn the skin.

8.3 A supply of filters may be prepared ahead of time and kept in the oven until needed. After drying, handle dish and filter with forceps or tongs only, never the fingers. Place in a desiccator for 20 ± 5 min and then weigh the filter and the pan. Weigh, to the nearest 0.1 mg, immediately before use.

8.4 Assemble membrane filter apparatus with glass filter in place and start suction. Apply a small amount of water to seat filter against the support.

DANGER: Damaged vacuum flasks can implode under high vacuum. Inspect flasks before use for visible signs of cracks, chips, or other signs of abuse. Do not use damaged or injured flasks.

NOTE: Glassware can become "bruised," or weakened by abuse, such as from impact. Discard any vacuum flasks if they have suffered any abuse.

NOTE: Flasks utilizing 'webbing,' tape, or plastic coating is encouraged so that any flying glass would be contained in event of an implosion. (The nature of the liquid being filtered may make this impractical.)

8.5 Shake the sample vigorously and rapidly transfer 100 mL to the graduated cylinder. Record volume to the nearest 1 mL if greater than 20 mL of sample is taken. Pour measured volume through the tared filter.

NOTE: If suspended matter is low, a larger volume may be filtered. With some samples, such as some pulp mill effluents, 100-mL aliquot causes filter plugging and prolonged filtration times. If filtration of 100 mL of sample is not accomplished within a reasonable time (three min), the sample volume should be reduced to allow for filtration within this time frame.

For NPDES, filter smaller increments of the sample and record the time necessary for filtering. Choose the proper sample volume to ensure that filtration is completed just short of the time a significant decrease in filtration rate occurs.

NOTE: For original volumes of less than 10 mL, dilute 10 ± 0.5 mL of the sample to 100 mL or more in volumetric flasks. Run the test on the diluted sample.

8.6 Rinse the graduated cylinder and filter holder three times with 10 ± 2-mL volumes of water. Allow the washing to completely pass through the filter before beginning the next.

8.7 Release the vacuum.

8.8 Remove the filter from the membrane filter assembly, being careful not to lose any of the sample, and place in the tared aluminum pan. Discard filtrate down the sink.

8.9 Dry 4 to 24 hr at 104 ± 1°C. Cool in a desiccator for 20 ± 5 min and weigh the pan and filter.

WARNING: Skin contact with hot surfaces may burn the skin.

8.10 Samples are dried overnight and historical data has shown that this is sufficient to reach a constant weight. If a shorter drying time is used or at client request, repeat step 8.9 to make sure there is less than 0.5 mg change of mass in the filter. Record the service request number for this information in the Laboratory Standard Recovery notebook.

8.11 Run one blank, a control, and one sample in duplicate in conformance with laboratory guidelines, with a minimum of one of each of these per batch or every 10 samples, whichever is more frequent. The blank is done without running any water through the filter except for the initial rinse.

The blank generally has a loss of 0.1 mg, which is <u>not</u> subtracted from the samples. If the blank gains mass or loses more than 0.2 mg on a 47 mm filter, the samples should be redone.

The controls shall meet the established QC requirements for that control sample. The manufacturer supplies acceptance limits with each lot of standard. Duplicates shall meet the QC requirements for that type of sample.

NOTE: The current control is NSI Standard Reference Material. The recovery limits are 85-115%.

9.0 QUALITY ASSURANCE

Quality assurance is addressed in part 8.11 above.

10.0 REPORT

10.1 Calculations:

Calculate suspended solids as follows:

$$\text{Suspended solids, mg/L} = \frac{(A - B)(1{,}000{,}000)}{C}$$

Where:

A = Mass of filter/pan and residue in g.
B = Mass of filter/pan in g.
C = Volume of sample filtered in mL.

10.2 Do not report more than 3 significant figures.

10.3 Results should be reported in mg/L.

10.4 The lower detectable amount of material is dependent upon the amount of the initial starting volume. The mass of residue used to calculate the detection limit is 1mg.

Example: (0.0010g) (1,000,000) ÷ 500mL = 2 mg/L
2 mg/L would be the detection limit for a 500-mL aliquot.

10.5 Precision and Accuracy

There is no way to determine the precision and accuracy for this method since it generates a method defined value. There is also no defined standard with which to make an objective determination of TSS recovery.

The precision of the determination varies directly with the concentration of suspended solids and degree of difficulty in obtaining a representative sample for analysis. Analysis of typical Weyerhaeuser wood products and pulp mill waste streams by WATS Laboratory showed a standard deviation of ± 1 mg/L (14 % - 11 replications) at 7 mg/L, ± 2 mg/L (4 % - 6 replicates) at 52 mg/L, and ± 3 mg/L (3% - 12 replications) at 100 mg/L.

The results of twenty 94.2 mg/L TSS standard run over a period of 7 months (Oct. 2006-April 2007) gave a recovery of 92.2 mg/L with a standard deviation of 2.1 mg/L.

11.0 KEY WORDS

glass fiber, filter, non-filterable solids, solids, suspended solids, total suspended solids, TSS, water, wastewater

12.0 REFERENCES

12.1 Standard Methods for the Examination of Water and Wastewater, 18th ed., p. 2-56, Method 2540D.

12.2 EPA Manual of Methods for Chemical Analysis of Water and Wastes, March 1983, p. 160.2.

12.3 "Precision of the TSS Test," Weyerhaeuser Technical Report by S. Vincent, February 1977.

12.4 ASTM method D5907.

13.0 REVISION HISTORY

13.1 7/13/07 8.9 – changed the drying time; 8.10 - changed wording to eliminate requirement to check constant weight; 8.11 – added information about QC requirements; 10.5 – Updated precision and accuracy

1.0 SCOPE

1.1 This Standard Operating Procedure (SOP) is written to provide guidance for training laboratory personnel in instrument operations and testing methods and for documentation of that training.

1.2 A copy of this procedure is to be present for the trainer and trainee at the bench at time of training. This is necessary so that a procedure is performed in a proper and consistent manner.

1.3 This procedure is referred to in the Quality Manual, Section 4.6.4.b.

2.0 SUMMARY OF THE PROCEDURE

The purpose of this SOP is to familiarize the trainee with lab safety needs before starting any work in the laboratory. The trainee will be:

1. Familiarized with the method or procedure before starting any work in the laboratory. The trainer will demonstrate the entire procedure to the trainee.

2. The trainee will then perform the entire procedure under the direct supervision of the trainer. The training will continue until the trainee has generated valid data (for a test method) or demonstrated proficiency to the satisfaction of the trainer (for a standard operating procedure).

3. Acknowledgment of revisions and modifications to a procedure will be documented. An employee who has been qualified to perform a procedure and who has been doing so for an extended period of time will have this proficiency documented in lieu of training.

3.0 PROCEDURE

3.1 Weyerhaeuser Analytical Sevices Laboratory Chemical Hygiene Plan

Before any work can be performed in the laboratory by any employee, the employee must be familiarized with the Weyerhaeuser Analytical Sevices Laboratory Chemical Hygiene Plan.

3.1.1 The trainee must read the Weyerhaeuser Analytical Sevices Laboratory Chemical Hygiene Plan.

3.1.2 The trainee must then have a discussion with the Chemical Hygiene Officer for Analysis and Testing Services. This discussion will be general with regard to the plan and specific with regard to the area in which the trainee will be working.

3.2 The Training Form

3.2.1 All the training forms can be found by clicking on 'Forms and Templates' on the A&T Homepage on the Web.

3.2.1.1 The 'TrainRecord - Methods' form is used when an employee is learning a test method that is new to the employee.

3.2.1.2 The 'TrainRecord – Processes' form is used when an employee is learning a standard operating procedure that will not be used to generate data.

3.2.1.3 The 'TrainRecord – ProcedMod' form is used when an employee is receiving refresher training on a test method (for which the employee has been previously trained and for which the employee has already generated acceptable data) or demonstrated sufficient proficiency on a standard operating procedure.

3.2.1.4 The 'TrainRecord – PreviousQual" form is for an employee who has been performing a test method or standard operating procedure for an extended period of time (3 years or more) or who has developed the method or procedure. That employee is considered to be a 'grandfather' with respect to that method or procedure. A signed document stating that the employee had been previously qualified is sufficient to document the training. Proficiency must be documented. See 3.5.

3.3 Training an Employee who is learning a new test method or standard operating procedure

3.3.1 The trainee will read the test method and/or standard operating procedure. A discussion of the test method and/or standard operating procedure with the trainer will follow to be sure there are no questions with regard to the method or procedure and that the trainee has a good understanding regarding any hazards or dangers involved in performing the method or procedure.

3.3.2 The trainee will fill out the first two portions of the 'TrainRecord - Methods' form for a test method and/or the first two portions of the 'TrainRecord – Processes' form for a standard operating procedure.

3.3.3 The trainer will demonstrate the procedure from start to finish as the test method or standard operating procedure dictates. The trainer and trainee will sign the third portion of one of the 'TrainRecord' forms, entitled 'Demonstration'.

3.3.4 A secondary trainer will observe the trainee perform the procedure for a different SR when a secondary trainer is available, giving advice and guidance where needed. The trainer and trainee will sign the fourth portion of one of the 'TrainRecord' forms, entitled 'Observation'.

3.3.5 Training is complete for a test method upon production of acceptable Data/Results. The date of the data generation (completion) and service request number or IPAR (Initial Precision and Accuracy Record) are recorded. The technical specialist in charge of this particular test method will sign the 'TrainRecord - Methods' form after examining the data. Training is complete for a standard operating procedure when the trainer believes the necessary criteria and/or proficiency is met.

3.3.5.1 For environmental regulatory work, initial demonstration of capability, if required by the method, shall be kept as part of the training file. See Section 3.7.

3.3.5.2 For wet lab work, demonstration of capability is complete when the trainee successfully generates "Grande Prairie Control" data as it applies to refining, CSF and sheeting. A copy of the Grande Prairie Control data generated is attached and shall be kept as part of the training file when applicable.

3.3.5.3 For physical test work, demonstration of capability is complete when the trainee successfully generates "collaborative testing" and calibration testing data. A copy of the collaborative testing data generated is attached and shall be kept as part of the training file.

3.3.6 The form is then sent to the Quality Advisor for further examination and signature. Training records are then filed with the appropriate Quality Advisor.

3.4 Training an Employee to perform a test method or SOP after the test method or SOP has been updated

3.4.1 The degree of updating is important. The technical specialist must determine if a new training/IPAR is necessary. If a new training/IPAR is not necessary, then the revisions must be reviewed by the employee and documented in the 'Description of Changes' section of the 'TrainRecord – ProcedMod' form. The employee will sign and date the form in the appropriate place certifying that he/she has read, understood and agreed to perform the most recent version of each procedure listed.

3.4.2 If a new training/IPAR is necessary, these results must be documented.

3.4.3 The owner of the particular procedure (Technical Specialist, Project Manager, Operations Manager, Laboratory Manager or Quality Advisor as applicable) will then sign and date the form in the appropriate place.

3.5 Documenting the Qualifications of an Employee Qualified Long Ago

An employee who has been qualified to perform a procedure and who has been doing so for an extended period of time will have this proficiency documented in lieu of training on the 'TrainRecord – PreviousQual" form. The IPAR must be performed no matter how long an employee has been doing the test. While an employee might be able to do the method and have no training record, the employee must demonstarte the method can be done well.

The employee will list all procedures for which he/she is previously qualified with the procedure number, procedure name and the year of the date at which time the employee started performing this particular procedure.

3.6 Completion of Training

Training is not complete until the training records are filed with administrative personnnel. Until training is complete, the trainee will be directly and closely supervised by the trainer.

3.7 Training for Environmental Regulatory Work

Precision and accuracy must be demonstrated by the analyst before prepping, extracting or analyzing any samples.

3.7.1 A clean blank sample of the matrix must first be generated. Four lab control samples are then generated. Each sample is spiked with the surrogate solution as dictated by the method. Four lab control samples are spiked with a lab control spike solution made up of each single component

parameter of interest. The blank sample as well as four lab control samples are extracted and analyzed. The blank sample and lab control samples should be carried through all stages of the sample preparation and measurement. . The blank sample and lab control samples must meet the precision and accuracy requirements stated in the method. If the method does not have these P&A requirements, then acceptable values will be determined by the LCS data of previously run batches of samples.

4.0 REVISION HISTORY

10/3/03: Added "when possible" to section 3.3.4. Fixed typos. 05/31/05: Modified 3.3.4 and 3.3.6.